Cationic Pretreatment for Reactive Dyeing of Cotton and its Simultaneous Antibacterial Functionalisation

Abstract

Reactive dyes are chemically bonded to a cotton fibre surface. The anchor groups of dye molecules initiate this covalent bonding. In addition to this anchor group, reactive dyes also contain charged functional groups that are often negatively charged sulphonate groups –SO₃⁻. These negative groups are part of the dye to enable its solubility in water. In industrial applications, dyes are applied as part of a water-based dye bath. The aim of the presented study was to improve the dyeing of cotton through the cationic modification of the textile, supporting an attraction to negatively charged dye molecules. In this way, the dye up-take and achieved colour depth should be improved. The current study was performed with a vinyl sulfone reactive dye. Three different nitrogen containing cationic organic substances were used for cotton pretreatment. In addition to colour properties, the antibacterial properties of prepared textile samples were also studied because antibacterial properties are often related to compounds containing amino and ammonium groups. Finally, it was shown that the cationic pretreatment with two of the three studied agents increased the dye up-take of cotton fabric from the dye bath. At the same time, one cationic agent can introduce antibacterial properties to treat cotton fabrics against two different types of bacteria: E. coli and S. warneri. The simultaneous application of a functional property during an optimised dyeing process was demonstrated in this case and can serve as an example for further applications.

Keywords: Coloration, dyeing, antibacterial, cationic compounds

Izvleček


Ključne besede: obarvanje, barvanje, protitabakterijski, kationske spojine

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1 Introduction

The dyeing of cotton can be performed using various dyes from different dyestuff categories, i.e., as vat dyes, direct dyes or reactive dyes. Among all dyestuffs, reactive dyes are supposed to lead to the best wash fastness of coloration on cotton fabrics. However, it should be kept in mind that one problem of dyeing of cotton with reactive dyes is the hydrolysis of the reactive dye in the dye bath. In simple terms, a reactive dye can be understood as a dye molecule containing three parts with different functions. These are the chromophore, the reactive anchor and charged ionic groups. While the chromophore is responsible for coloration, the reactive anchor supports covalent bonding between dye molecules and the cotton fibre surface. The ionic groups result in solubility in a water-based dye bath. In acid dyes, anionic charged groups are also responsible for the attractive interaction between the acid dye and cationic wool or polyamide fibres. Wool and polyamide fibres contain a positive net charge due to the protonation of containing amino groups. Acid dyes contain a negative charge. For this reason, wool and polyamide fibres support a certain electrostatic attraction to the acid dye, thus supporting the dyeing process and dye fixation. The ionic attraction of anionic groups in reactive dyes to positively charged groups can be analogously used to improve the dyeing performance of reactive dyes [1]. For this purpose, cationic functional groups must be introduced to the cellulosic structure of the cotton fibre. A cotton fabric treated with cationic agents obtains positive charges on the cotton fibre surface. If a reactive dye containing anionic groups is then applied to this cationised cotton, the dyeing process can be supported analogously to the dyeing process of wool using an acid dye. In literature, many different procedures are described for the introduction of cationic groups to the cotton fibre surface.

Quaternary ammonium compounds are fixed to cotton using an epoxy anchor. For subsequent dyeing with acid dyes and reactive dyes, improved dyeing properties were observed [2]. In these experiments, the increased dye up-take was directly correlated to the amount of previously applied quaternary ammonium groups [3]. Instead of an epoxy anchor, the quaternary ammonium compound can also be anchored to cotton using a chlorine hydroxyl propyl group [4]. In this way, an ionic attraction between dye molecules and cationised cotton was introduced. Improved colour-depth can be achieved for the application of different reactive dyes [4]. In addition, polymers with quaternary ammonium groups can be used for cotton pretreatment [5]. In an example by Blackburn et al., quaternary ammonium groups were part of an aliphatic ring system attached to the backbone of the polymer. Here, an increase in colour depth after dyeing with reactive dyes was also observed [5]. As a special cationic polymer, commercially available cationic starch can also be used for the modification of cotton [6, 7]. The application of this cationic polyelectrolyte improves the dye-fibre interaction. The improved dye up-take in this case is caused by the presence of cationic groups and the increased fibre roughness as the result of the applied starch [6]. In addition to these polymers with quaternary groups, polymers with amino groups can also be used for the modification of cotton. For this purpose, the chloride salt of polyvinylamine is used to improve the dyeing process of cotton with reactive dyes [8]. A cationic pretreatment is also used to improve the dyeability of cotton with natural dyes [9, 10]. If natural dyes contain negatively charged functional groups or groups that can gain a negative charge easily through deprotonation under moderate alkaline conditions, they probably show an electrostatic attraction to positively charged cationised cotton fibres, as well. For this reason, the dyeing process is improved. This phenomenon is probably the same for the application of a negatively charged reactive dye on cationised cotton textiles.

In addition to the influence of dyeing properties, quaternary ammonium containing cationic compounds are often mentioned for their antibacterial effects on textile substrates [11-15]. A special type of antibacterial active quaternary ammonium compound is based on the cationically modified nitrogen component DABCO (1,4-Diazabicyclo(2.2.2)octan), which is a cyclic nitrogen compound [16]. For the achievement of antibacterial effects, not only the antibacterial effects of quaternary compounds have been reported; other nitrogen containing compounds such as PHMB (polyhexanid) have also been reported [11]. The antibacterial effect of PHMB is related to the presence of an amino group attached to the polymer structure. Another prominent antibacterial polymer containing amino groups is bio-based...
chitosan [17-19]. The antibacterial activity in such cases is often related to the acidic conditions of the surrounding medium due to the necessary protonation of the containing amino group [20]. In addition to bio-based chitosan, synthetic polymers containing an amino group are also known for their antibacterial activity. A prominent example in this area are dendrimers with terminated amino groups [21]. Reactive dyes equipped with cationic groups can be used to introduce antibacterial properties to cellulosic fibres. In this reported application, coloration and antibacterial functions are achieved at once through the application of a single compound [22]. As an alternative to cationic nitrogen compounds, simple amino compounds can also be used to introduce antibacterial properties to textiles. An example is the application of polyvinylamine for the functionalisation of fabrics made from high-performance polyethylene [23].

With this background, the purpose of the presented study was to evaluate different cationic pretreatments for cotton with the aim of simultaneously improving dyeability with reactive dyes and achieving antibacterial properties. For the actual evaluation, three different commercially available cationic substances were chosen and applied in increasing concentrations onto cotton substrates. The dyeability of modified cotton was tested through the application of a vinyl sulfone reactive dye that contained two anionic groups. The antibacterial properties were tested against two different bacteria before and after the dyeing process. The achieved results were promising and showed that a cationic pretreatment can be used for simultaneous and different modifications of cotton fabrics.

2 Experimental section
2.1 Materials and sample preparation
For all sample preparations, a plain weaved cotton fabric with a weight of 150 g/m² was used. This cotton fabric was treated with three different cationic agents applied in three different concentrations to determine their influence on the subsequent application of a reactive dye. These cationic agents were RUCO-PUR SEC, supplied by Rudolf GmbH (Geretsried, Germany), RUCO BAC HSA, also supplied by Rudolf GmbH (Geretsried, Germany), and PERFIXAN F 5000, supplied by Textilchemie Dr Petry GmbH (Reutlingen, Germany). All these chemicals were supplied as water-based solutions and further diluted with water as recommend by the suppliers. The aqueous cationic agent RUCO-PUR SEC was named the hydrophilic agent. It is cationically active and based on polyurethane and silicone compounds. This agent was further diluted with water to a concentration of 30, 45 or 60 g/L. The pH value was adjusted to 4.5 by adding acetic acid. The agent RUCO BAC HSA is an aqueous solution of the quaternary ammonium compound - dimethyltetradecyl (3-(trimethoxysilyl)propyl) ammonium chloride – and was distributed as the cationic antibacterial agent. This agent was further diluted with water to concentrations of 2, 11 or 20 g/L. The pH value was adjusted in the range of 4.5 to 5.0 by adding acetic acid. The agent PERFIXAN F 5000 is described as precationising agent recommended for denim articles and for dyeing procedures with anionic dyes. It is an aqueous solution of a polyamine component containing a pH 2.5 to 3.5. For application, this agent was further diluted with water to a concentration of 20, 40 or 60 g/L. Of course, cotton fibres can be damaged under strong acidic conditions, but the agent used, PERFIXAN F 5000, was not applied as a pure substance. It was applied in concentrations of between 20 to 60 g/L after dilution with water. The acidity of the applied agent was decreased by this dilution, so potential damage to cotton fibres was minimised. The used concentrations for these studied cationic agents were based on the recommendation of the suppliers of these chemicals. All cationic agents were applied in an HFR 46292 padding machine supplied by Werner Mathis AG (Oberhasli, Switzerland). After application, drying was performed at 140 °C for 60 seconds using an OHE 4408787 lab dryer supplied by Werner Mathis AG. For the production of a reference sample, analogous treatment by padding the cotton fabric with pure water was carried out.

Dyeing was performed using the Reactive Black 5 (RemazolBlack B; CAS 17095-24-8) dye with the sum formula of C_{26}H_{12}N_{3}Na_{4}O_{19}S_{6} and a molecular weight of Mw 991.8 g/mol. The company Clariant (Frankfurt am Main, Germany) supplied this dye. For dyeing purposes, an aqueous dye bath containing the following components was used: Reactive Black
dye (5.3 g/L), NaOH (0.68 g/L), Na₂SO₄ (65 g/L) and Na₂CO₃ (25 g/L). To prepare the dye bath, 3 g of Reactive Black dye was first dissolved in 70 mL of hot water. After that, 65 g of Na₂SO₄ was added and dissolved. Then 25 g of Na₂CO₃ was added and dissolved. Finally, 0.68 g of NaOH was added and dissolved, and the vessel was filled with water until 1 L was reached. The bath ratio was set to 1:10 (1 g fabric to 10 g dye bath). The concentration of the used reactive dye was 5.3 g/L or 0.53 weight-% in relation to the volume of the dye bath. In relation to the amount of treated cotton textile, the amount of dye was 5.3 weight-%. The dyeing process was performed in an Ahiba Polymat (model PM10) dyeing machine. The process temperature for dyeing was 60 °C, which was applied for 100 minutes, followed by heating for 10 minutes. After the dyeing procedure, the fabrics were rinsed with cold water, followed by a washing cycle at 95 °C for 5 minutes. That washing was performed in an S014.95 washing machine supplied by Werner Mathis AG. At the end, the fabrics were line-dried at room temperature.

2.2 Analytics

The remaining dye concentration in the dye bath after the dyeing process was determined. For this purpose, samples were taken from the dye bath after the dyeing process was completed. A total of 1.5 g of the remaining dye solution was taken and diluted with 8.5 g water. The absorption spectra of this solution were determined using a UV-2600 photo spectrometer from Shimadzu (Japan) in an arrangement of direct transmission. The coloration of prepared dyed textiles was studied using the same photo spectrometer with an integrated sphere and by measuring diffusive reflection. The colour properties were recorded as K/S-spectra. In order to determine K/S-spectra, the reflection spectrum was first measured in an arrangement of diffusive reflection. This reflection spectrum was transferred using the Kubelka-Munk function into a K/S-spectrum, which demonstrated the absorption of light for the studied textile sample as a function of the wavelength of light. A barium sulphate plate was used as white reference material for these reflective measurements. In addition, the difference in the colour strength of the dyed fabrics with cationic pretreatment was determined using a Datacolor 400 colorimeter with a D65 light source (Datacolor, Luzern). For these measurements, the fabrics were folded into four layers. As a reference sample, the dyed cotton fabric without cationic pretreatment was used and set to a value of 100%. The FT-IR spectra of cotton samples were recorded using an Excalibur 3100 IR-spectrometer (Varian Inc.).

Bacterial viability was determined by using 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT) as previously reported [24]. In brief, E. coli (strain BL21(D3)) and S. warneri (strain dsm-20316) were cultured in a Luria–Bertani (LB) medium or in a Trypticase Soy Yeast Extract (TYSE) medium, respectively. For the experiments, 200 μl bacterial suspensions (1:250 dilution of an overnight culture) per cavity were seeded in sterile 96-multiwell cell culture plates (Techno Plastic Products AG, Trasadingen, Switzerland). Cells were grown in the presence of textile samples (circles with 5 mm diameter prepared using a conventional hole puncher) for 3 hours at 37 °C and rotated at 250 rpm in a PST-60-HL-4 orbital shaker (BioSan, Riga, Latvia). The cells were then incubated with MTT (final concentration 0.1 mg/l) in their respective culture media for 5 minutes, lysed in isopropanol for an additional 30 minutes, and their viability determined by measuring absorption at 570 nm with a reference wavelength of 700 nm on a Tecan M200 multiwell-plate reader (Tecan, Crailsheim, Germany). Control viability was measured in a test arrangement in the absence of any textile fabric, but otherwise identical to the other samples and set to 100%. The absorption determined with the same setup in the absence of bacteria was set to 0%. The antibacterial tests were repeated four times for each sample. The average of the repeated measurements was given as the remaining bacterial viability.
3 Results and discussion
3.1 Preliminary investigations before dyeing

As preliminary experiments before dyeing, FT-IR spectroscopy and UV/Vis spectroscopy were performed on untreated cotton fabrics and on fabrics after application of the cationic agent. FT-IR spectroscopy was performed to determine whether the applied cationic agent can be detected on cotton fabrics. The measured FT-IR spectra are shown together in Figure 2. The determined spectrum from the untreated cotton fabric exhibited a similar shape compared to cotton spectra reported in literature [25]. The prominent peaks were from the strength vibrations of the C-O bond, C-H bond and the O-H, with maxima at the wavenumbers 1053 cm\(^{-1}\), 2897 cm\(^{-1}\) and 3329 cm\(^{-1}\). The FT-IR spectra of samples after application of cationic agents were prepared for preparation with the highest applied concentration of these agents. For the RUCO BAC HSA agent, a nearly similar FT-IR spectrum compared to cotton was recorded. It is thus not possible to detect this agent on cotton using this spectroscopic method. The RUCO BAC HSA agent contained a quaternary ammonium group. This group was related to C-N bonds with stretch vibrations in the range of 1020 to 1220 cm\(^{-1}\) [26]. However, an especially strong signal from the cotton substrate with the C-O vibration also appeared in this region. It is thus probable that the signal from the cotton substrate covered the signal of the added RUCO BAC HSA agent. For the RUCO-PUR SEC agent, a nearly similar FT-IR spectrum compared to cotton was recorded. Only one weak peak in the fingerprint area at 779 cm\(^{-1}\) was identified and not detected in the pure cotton fabric. According to literature, this peak can be caused by a Si-C vibration in an OSi-CH\(_3\) group [26]. This result is in line with supplier information that this agent also contains silicone compounds. For the PERFIXAN F 5000 agent, the peak at a wavenumber of 1639 cm\(^{-1}\) exhibited an increased intensity, while a new peak with a weak intensity appeared at 1543 cm\(^{-1}\). This agent was based on polyvinylamine and contained amine groups that were related to C-N bonds with stretch vibrations in the range of 1020 to 1220 cm\(^{-1}\) and N-H bonds with stretch vibrations close to 3335 cm\(^{-1}\) [26]. However, both areas were covered by several strong vibration signals of the cotton substrates itself. The stronger signal at 1639 cm\(^{-1}\) and the new signal at 1543 cm\(^{-1}\) can be explained by the presence of amide groups in this agent. Polyvinylamine was prepared through the degradation of polyacrylamide [27, 28]. If this degradation is not complete, there would be remaining amide groups in this polymer that could be detected using IR-spectroscopy.

To evaluate the colour properties of textiles after the dyeing process, the K/S-UV/Vis spectra of undyed cotton fabrics with and without cationic treatment were determined (Figure 3). This measurement was done to determine whether the applied cationic agents can affect the colour properties of the treated cotton fabrics by themselves. All these undyed cotton fabrics exhibited low K/S-values in the range of visible light (400 nm to 750 nm) of only 0.1. These textiles were mainly uncoloured and the cationic treatment did not affect coloration. In the range of visible light, the K/S-values were very low and did not show significant differences between the treated and untreated textiles.
UV-light, the cotton treated with PERFIXAN F 5000 exhibited a significant maximum at around 320 nm. This maximum was probable related to aromatic structures in the applied cationic component.

### 3.2 Dyeing properties and coloration results

Before determining the colour properties of dyed fabrics, the absorption spectra of the dye bath after the dyeing processes were measured (Figure 4). This measurement was taken to determine the remaining dye in the dye bath and for the dye not taken up by the dyed cotton fabric. The determined absorption spectra were compared with a reference spectrum achieved for an analogous dyeing procedure performed with the same cotton fabric, but without any cationic treatment. Absorption spectra with lower absorption values compared to this reference spectrum indicated an increased dye up-take from the dye bath by the cationised cotton. Different results were obtained for the three different types of cationic pretreatment (Figure 4). Treatment with the RU- CO-PUR SEC agent led to a lower applied concentration of cationic agent and to higher absorption values in the remaining dye bath. Only in the case of the highest applied concentration of 60 g/L RU- CO-PUR SEC was nearly the same absorption as the reference observed. For this reason, no improvement of dye up-take could be achieved in the applied dyeing procedure with RU- CO-PUR SEC. In contrast, a significant decrease in dye concentration in the remaining dye bath was achieved with cotton pretreatment using RUCO BAC HSA (intermediate or high concentration) or using the PERFIXAN F 5000 (with all concentrations) agent. Here, it is probable that the up-take of dye by the cationised cotton increased. The up-take of the reactive dye was significantly affected by the type of cationic treatment performed on the cotton fabric in advance.

The colour properties of dyed cotton fabrics were determined as colour intensity (Figure 5) and K/S-spectra (Figure 6). The colour intensity was given as a percentage in relation to the coloration of cotton dyed without previous cationic treatment. This reference value was set to 100% (Figure 5). The determined colour intensity was seen as a function of the concentration of applied cationic agents. Treatment with the RU- CO-PUR SEC agent led to the lower coloration of cotton fabric compared with the dyeing procedure using untreated cotton. Increasing the concentration of RU- CO-PUR SEC also led to an increase in colour intensity. However, even with the highest concentration for pretreatment with RU- CO-PUR SEC, the same colour intensity as the reference untreated cotton sample could not be achieved. These results are in line with the remaining high dye concentration in the remaining dye bath. It can thus be said that this cationic agent cannot be used to improve the dyeing procedure for the studied reactive dye on cotton. In fact, this result is in some way surprising because the cationic active RU- CO-PUR SEC agent should have improved the up-take of the applied reactive dye. A possible explanation could be that the number of cationic sides in this agent was not as high as the other studied additives. Thus, the effect on dyeing behaviour with the studied reactive dye is not strong.

In contrast, the coloration result of subsequent dyeing improved with pretreatment using the other two cationic agents. For both products, the colour intensity increased as a function of the concentration of the cationic component used for pretreatment. However, the type of increase was different (Figure 5). For the RUCO BAC HSA agent with the lowest concentration, an initial decrease in colour intensity was identified. The colour intensity was then continuously increased by increasing the concentration of this agent. For the PERFIXAN F 5000 agent, pretreatment with a medium to high concentration led to only a minor subsequent increase. A kind of plateau value could thus be estimated.

The evaluation of the K/S-spectra of the dyed samples completed this picture (Figure 6). The K/S-spectra for dyed samples after RU- CO-PUR SEC pretreatment were very similar to the reference spectrum of a sample without any pretreatment. Here, almost no change in coloration was caused by the cationisation of cotton with this type of cationic compound. Using a pretreatment of RUCO BAC HSA, a significant increase in the K/S-values using the cationic treatment was observed. The reference sample exhibited a maximum K/S-value of 17.0 at 606 nm. The samples with RUCO BAC HSA pretreatment achieved a maximum K/S-value of 19.4 at 597 nm. Treatment with PERFIXAN F 5000 also resulted in a high K/S-value of 19.7. However, there was also a stronger significant shift in the position of the maximum to a wavelength of 590 nm. This shift indicated that not only colour intensity but also colour shade changed as the result of the applied cationic treatment.
Figure 4: Absorption spectra of dye baths after the dyeing process. Compared are the spectra of dye baths after the dyeing of an untreated cotton reference and cationised cotton fabrics. The amount of applied cationic agent for the pretreatment of cotton is directly observable in the graphs. In order to record the spectra, the dye bath was diluted with water in a ratio of 15:85.

Figure 5: Colour intensity of dyed cotton fabrics with different pretreatments in relation to a reference cotton fabric dyed without any pretreatment. The colour intensity is shown as a function of the concentration of the cationic agent used for pretreatment.
3.3 Antibacterial properties

Antibacterial properties were studied against two types of bacteria: *E. coli* and *S. warneri* (Figure 6). The remaining bacterial viability was shown as a function of an increase in the concentration of the applied cationic agent. For all samples, bacterial viability was compared before and after the dyeing process (Figure 7). A value of 100% for bacterial viability represented the reference testing procedure without the addition of any textile sample.

In the presence of the pure cotton sample without any further treatment or dyeing, a remaining bacterial viability for *E. coli* of 79% and for *S. warneri* of 80% was determined. In the case of dyeing cotton without a cationic agent, values for the bacterial viability for *E. coli* of 75% and for *S. warneri* of 74% were observed. This small decrease in viability after dyeing was in the range of the standard deviation of this test arrangement. The applied dye thus had no probable antibacterial effect.

Compared with these reference measurements without a cationic agent, the effect achieved with cationic treatment was different, depending on the type of cationic agent applied (Figure 7). With the application of even the highest concentration of the PERFIXAN F 5000 agent, no decrease in bacterial viability was identified. This agent exhibited no clear antibacterial properties. According to supplier information, the Perfixan agent is related to the chemical structure of polyvinylamine. As a result, this agent is supposed to contain a large number of amino groups. Such amino group containing compounds often contain antibacterial properties. These antibacterial properties are related to an acidic medium leading to the protonation of the amino groups. However, the antibacterial tests were performed under neutral conditions. It should also be clear that amino containing compounds can be antibacterial, while not all chemicals containing amino groups are antibacterial. Good examples of amino containing groups without antibacterial properties are ordinary amino acids and proteins.

An effect against *S. warneri* was observed with the RUCO-PUR SEC agent at the highest applied concentration. In this case, a bacterial viability of only 27% was identified. That value then rose to 60% after the dyeing was performed. This antibacterial effect was clearly seen, but was not very strong. The loss in antibacterial activity after dyeing can

![Figure 6: K/S-spectra of dyed cotton fabrics. The spectra are related to different pretreatments of the dyed cotton with different chemicals in increasing concentrations. The observed reference spectrum is for a cotton fabric without any cationic treatment.](image-url)
be explained by the removal of some of the cationic agent during the dyeing process. If the RU-CO-PUR SEC agent was applied in the lowest concentration of 30 g/L, a small increase in bacterial viability for the bacteria *S. warneri* was observed. This small increase in bacterial viability with the lowest RU-CO-PUR SEC concentration was in the range of standard variation for this measurement and did not indicate that this agent could support the growth of bacteria in low concentrations.

In the presence of the cationic RU-CO BAC HSA agent, the antibacterial effect was significant against both tested bacteria types: *E. coli* and *S. warneri* (Figure 7). That activity was particularly strong against *S. warneri*. This was shown by a remaining bacterial viability of less than 0.5%. This strong effect should be expected because this agent is promoted by the supplier as antibacterial finishing agent. The antibacterial effect decreased when the dyeing process was performed. However, the effect is still excellent, even after the dyeing procedure, especially against *S. warneri*. Such a decrease in antibacterial activity after dyeing can be explained by an insufficient fixation of the cationic agent on the cotton surface. For this reason, the RU-CO BAC HSA agent is probably removed in part from the cotton samples during the dyeing process, resulting in a decrease in antibacterial activity.

With these results, it can be said that these cationic agents can be used to achieve two effects with only one application. It is possible to simultaneously improve the dyeability of cotton for reactive dyes and to achieve antibacterial properties on the same cotton fabric.

4 Conclusion

In conclusion, it can be said that it is possible to introduce two advantageous properties to cotton in one step through pretreatment with cationic agents. Improved dyeability and an antibacterial activity can be achieved together through this simple application. It was also shown, however, that not all cationic agents are useful for improving dyeability or antibacterial effectiveness. The simultaneous application of a functional property during an optimised dyeing process was carried out and could serve as an example for further applications.

Figure 7: Antibacterial properties of differently pretreated cotton fabrics before and after the dyeing process. The antibacterial property is shown as the remaining bacterial viability as a function of the concentration of the cationic agent used for the treatment of cotton fabrics. The bacterial viability is shown as average of four independent measurements. The error bars indicate the standard deviation of this measurement.
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